Rapid radiocarbon (^{14}C) analysis of coral and carbonate samples using a continuous-flow accelerator mass spectrometry (CFAMS) system

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[1] Radiocarbon analyses of carbonate materials provide critical information for understanding the last glacial cycle, recent climate history and paleoceanography. Methods that reduce the time and cost of radiocarbon (^{14}C) analysis are highly desirable for large sample sets and reconnaissance type studies. We have developed a method for rapid radiocarbon analysis of carbonates using a novel continuous-flow accelerator mass spectrometry (CFAMS) system. We analyzed a suite of deep-sea coral samples and compared the results with those obtained using a conventional AMS system. Measurement uncertainty is <0.02 Fm or 160 Ryr for a modern sample and the mean background was 37,800 Ryr. Radiocarbon values were repeatable and in good agreement with those from the conventional AMS system. Sample handling and preparation is relatively simple and the method offered a significant increase in speed and cost effectiveness. We applied the method to coral samples from the Eastern Pacific Ocean to obtain an age distribution and identify samples for further analysis. This paper is intended to update the paleoceanographic community on the status of this new method and demonstrate its feasibility as a choice for rapid and affordable radiocarbon analysis.

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1. Introduction

[2] Radiocarbon has proved to be a powerful tool in many areas of ocean and environmental sciences. In paleoceanography and sedimentology, it is used to establish chronology over the past glacial cycle [*Balter*, 2006; *Fairbanks et al.*, 2005; *Hughen et al.*, 2004]. Past changes in ocean circulation and carbon cycle are reflected in the radiocarbon records of benthic and shallow corals [*Druffel et al.*, 2007; *Robinson et al.*, 2005]. Paleoclimate conditions in the western North Atlantic Ocean have been linked to coastal sediment cores dated using radiocarbon and sea surface temperatures [*Donnelly and Woodruff*, 2007]. The advent of accelerator mass spectrometer (AMS) methodologies greatly enhanced the use of radiocarbon in paleoceanography and, now, improvements in both sample preparation and AMS technology are further expanding its use.

[3] To date, AMS radiocarbon measurements have involved a significant number of preparative steps. This preparation is done to reduce post-depositional accretions or alterations, minimize the process blank and permit high precision measurements necessary for old and small samples. Although these chemistry steps are well established and performed routinely and efficiently, it can be argued that a significant portion of the effort, and hence cost, associated with an AMS measurement of these samples involves these preparation steps. For example, carbonate samples require an initial cleaning, followed by acidification to convert the carbonate to CO_2 gas [*Adkins et al.*, 2002]. The CO_2 is subsequently reduced to filamentous graphite by hydrogen reduction over iron [*Vogel et al.*, 1984]. The graphite is "packed" into an aluminum target and sputtered with a Cs ion beam in the source of an AMS system to produce a carbon ion beam from which the $^{14}C/^{12}C$ isotope ratio is determined. Radiocarbon ages down to about 50,000 radiocarbon years (Ryr) or 0.002 fraction modern (Fm) are routinely obtainable.

[4] Recently, reconnaissance dating has been introduced at the National Ocean Sciences Accelerator Mass Spectrometry facility (NOSAMS) to streamline the preparative steps and improve the cost effectiveness of radiocarbon analysis [*Burke et al.*, 2010]. It is intended for screening large samples sets at reduced precision so that patterns in processes such as ocean circulation and the carbon cycle can be identified. It is also used to minimize sampling risk by identifying samples that are suitable for further high precision analysis. Samples are combusted in an elemental analyzer and the resulting CO_2

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is converted to graphite using a sealed tube zinc reduction method [*Xu et al.*, 2007]. It has been successfully used to study the geographical and temporal distribution of benthic coral populations in the Southern Ocean [*Burke et al.*, 2010]. This study clearly demonstrated the usefulness of a rapid screening technique for radiocarbon to paleoceanography, and exposed its potential use in other disciplines such as sedimentology and paleo-ecology.

[5] To further facilitate analysis, the radiocarbon community has been developing gas ion sources (GIS) that directly accept CO_2 into the AMS system. The aim is to permit faster analysis of smaller and older samples by removing the time consuming graphitization step and reducing the potential for contamination during handling. Modified Cs sputter sources have been constructed and they function by passing sample CO_2 gas over a titanium insert fitted in the target of the ion source [*Bronk Ramsey and Hedges*, 1987; *Middleton et al.*, 1989; *Ruff et al.*, 2007]. These so-called hybrid gas ion sources are well suited to small samples (<200 μg) but are limited by factors including sample-to-sample memory, attainable precision and long analysis times. They have been coupled to instruments such as a gas chromatograph for compound specific radiocarbon analysis and are in routine use with elemental analyzers [*Bronk Ramsey et al.*, 2004; *Ruff et al.*, 2010; *Uhl et al.*, 2007].

[6] At NOSAMS, a novel gas accepting ion source has been developed that enables continuous-flow AMS (CFAMS) measurements of radiocarbon. Progressive development of the source has been reported by [*Roberts et al.*, 2007, 2011; *von Reden et al.*, 2011]. The system uses microwaves to generate a plasma from a constantly flowing CO_2 stream and a magnesium charge exchange canal to generate negative carbon ions for the AMS system. Sample memory compared to the hybrid gas ion source design is much less. However, the source requires a higher flow rate of gas and is less efficient than a hybrid source and thus, is ill- suited to small samples or high precision measurements. Nonetheless, direct acceptance of a continuous flowing stream of CO_2 has allowed analytical instruments such as a gas chromatograph to be interfaced directly to the CFAMS system for rapid and compound specific radiocarbon analyses [*McIntyre et al.*, 2009; *McIntyre et al.*, 2010].

[7] In this study we have developed an apparatus for the CFAMS system for radiocarbon analysis of carbonates, such as corals and limestone. The method is even more rapid and cost effective than the earlier reconnaissance dating method. The agreement, precision and repeatability of the results are assessed by comparing data with those obtained from a conventional AMS system using graphite. We then apply the method to benthic solitary corals taken from the Galapagos in the eastern Pacific Ocean to obtain an age distribution and identify samples for further analysis. The intention of this paper is to update the wider paleoceanographic community on the status of this new method and demonstrate its feasibility as a method of choice for rapid and affordable radiocarbon analysis.

2. Experimental

2.1. Standards and Samples

[8] Standards used in the study and their consensus radiocarbon ages and Fm values were: IAEA C1 marble, infinite radiocarbon age (0 ± 0.002); IAEA C2 Travertine, 7135 ± 5 (0.4114 ± 0.0003); University of California, Irvine, coral standard (CSTD), 450 ± 20 (0.9453 ± 0.0019); purified instrument grade CO_2 (reference gas), > Modern (1.0438 ± 0.0008). The C1 standard was used for the process blank correction and determination of background. C2 and CSTD were used as secondary standards.

[9] Initially, a survey of 20 deep-sea solitary scleractinian corals was undertaken to demonstrate the speed of the analysis, viability of the technique and obtain an age distribution to select corals for further analysis. Corals 1–18 were from the Drake Passage in the Southern Atlantic Ocean and corals 19–20 were from the New Seamounts in the Northern Atlantic Ocean [*Burke et al.*, 2010; *Robinson et al.*, 2005]. The corals had been previously analyzed (2007–2009) using the conventional AMS method [*Adkins et al.*, 2002]. The samples for rapid radiocarbon analysis were cut using a Dremel tool, physically abraded to remove visible contamination and cleaned with methanol [*Burke et al.*, 2010].

[10] A subset of 5 corals with a range of radiocarbon values from radiocarbon-dead to modern (0–1 Fm) plus the coral standard (CSTD) were selected for repeat analysis to assess accuracy, precision and repeatability. The coral samples were given an oxidative cleaning [*Burke et al.*, 2010] and were analyzed in triplicate.

[11] Finally, a set of 41 scleractinian corals of unknown age were analyzed to obtain a reconnaissance age distribution and identify samples for further analysis. The samples were collected from the Galapagos in the Eastern Pacific Ocean on cruise MV1007 with the *R/V Melville* from May 17 to Jun 18, 2010. Samples were given a methanol rinse and, along with the standards, were not acid etched to further speed analysis.

2.2. Sample Preparation for Rapid Radiocarbon Analysis

[12] Cleaned samples were acid etched, dried, ground, and 25–30 mg weighed into 7 mL vacutainers. Each vacutainer was evacuated through a 26 gauge syringe tip on a vacuum line to less than 10 mTorr and 1 mL of 85% H_3PO_4 added with a needle. Care was taken to ensure the integrity of the vacuum by covering the septum of the vacutainer with a layer of Apiezon M grease. The samples were heated in a water bath at 60°C until CO_2 was no longer being produced. Twenty-eight mg of $CaCO_3$ filled a vacutainer with CO_2 and thus, depending on the amount of sample, the vacutainers were slightly above or below atmospheric pressure. Each vacutainer was brought to atmospheric pressure via a syringe tip connected to Teflon tubing by either venting the excess CO_2 or, by drawing in degassed water that was prepared by sonication and He sparging. This step was done to ensure that when the vacutainer was pierced for sampling, that no argon was drawn into the vacutainer or, that no CO_2 was pushed back into the syringe. Reference gas was filled into evacuated vacutainers via a syringe tip connected to the outlet of the regulator.

2.3. CO_2 Gas Delivery Apparatus

[13] A schematic of the custom gas delivery apparatus constructed for this application is shown in Figure 1. Vacutainers were held in a laboratory rack and processed manually. CO_2 was displaced from the vacutainer using a

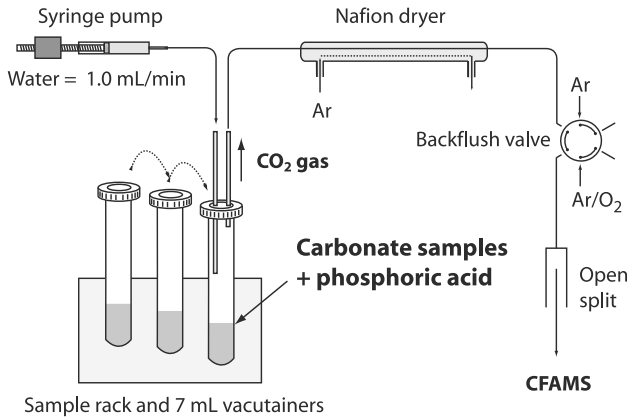

Figure 1. Schematic of the sample delivery apparatus.

dual needle arrangement. Degassed water was introduced into the vacutainer with a syringe pump at 1.00 mL/min via 1/16″ Teflon tubing and a 21 gauge syringe tip. The displaced CO_2 was pushed out through a second syringe tip connected to a 20 cm Nafion dryer with 0.36 mm I.D. tubing. The dry gas stream is passed to a 6–port Valco valve used for back flushing and into an open split where a constant flow was withdrawn by the ion source. The open split was a 10cm length of 3.2 mm O.D. × 1.6 mm I.D. Pyrex tubing. Connecting tubing was 250 μm fused silica tubing. The lines were backflushed for 10–30 s between samples. Argon with 10% oxygen was used to backflush the open split at 5 mL/min. Argon was used to backflush the sample line at 35 mL/min. A typical analysis sequence involved running reference gas, blank and secondary standards followed by 4 samples. The vacutainers could be filled up until the water reached the exit syringe tip leaving 0.5 −1 mL of gas.

2.4. AMS System

[14] The source, AMS system and performance have been described in detail elsewhere [*Roberts et al.*, 2007, 2010]. Briefly, CO_2 gas is introduced into a plasma chamber using a fused-silica capillary. A plasma is established using 2.45 GHz microwaves and a solenoidal magnetic field. Positive ions are extracted from the plasma using a triode system and negative ions are formed by charge-exchanging interactions with magnesium vapor. The resulting beam has a large energy spread and divergence. Thus, to avoid excessive transmission losses, an AMS system with large-gap optical elements and energy-stigmatic transport was custom built. Optimum intake of the source is approximately 0.37 mL/min of CO_2 resulting in a ^{14}C count rate of 400 cps at a total system efficiency of 0.21% (C atoms introduced/ions measured). For each sample, data were recorded for 5 min or until the CO_2 was exhausted to maximize the number of ^{14}C counts recorded.

2.5. Data Acquisition and Processing

[15] The three carbon isotopes were injected into the accelerator sequentially at a repetition rate of 10 Hz, and data from individual peaks were time sorted and statistically combined. Fraction modern ^{14}C values for the samples were calculated using the ion currents for ^{12}C and ^{13}C, and ^{14}C counts. Peaks were background subtracted and normalized to the reference gas. The values conform to standard reporting conventions [*Stuiver and Polach*, 1977]. When comparing two methods, data was plotted against each other and a linear least squares fit was performed with errors in both coordinates [*Reed*, 2010]. Where replicate data existed for a sample, a weighted-average and the error of the weighted-average was calculated prior to fitting [*Bevington and Robinson*, 2003]. To test if there was a statistically significant difference between the two methods, an unpaired, two-tailed, t-test for each coral using the triplicate data was performed assuming unequal variances.

3. Results

[16] Results for the initial survey, triplicate analysis and sample analysis are given in Tables S1, S2, and S3.[1] Summary statistical data for the combined blank and secondary standards are given in Table 1.

[17] For the initial survey, results of the least squares fit of the values previously obtained by the conventional AMS method (x) and the CFAMS values (y) yielded a trend line with an equation of y = 0.954(± 0.011)x + 0.006(± 0.003) and a Pearson correlation coefficient of 0.999.

[18] For the triplicate analysis of 5 corals plus the CSTD standard, the least squares fit of the values obtained by the conventional AMS method (x) and the CFAMS values (y) yielded a trend line with an equation of y = 1.008(± 0.008)x − 0.006(± 0.005) and a Pearson correlation coefficient of 0.999. The results of the t-test for the triplicate data from each coral gave P values greater than 0.05 in each case.

[19] Comparison of the CFAMS data from the initial survey (x) with the CFAMS data from the triplicate analysis (y) yielded a trend line with an equation of y = 0.9998 (± 0.007) x − 0.001(± 0.003) and a Pearson correlation coefficient of 0.999.

4. Discussion

4.1. Comparison of Methods

[20] The major differences between the methods described in this paper are shown in Table 2. The CFAMS method does not use graphitization and as a consequence, the time and cost of analyses are reduced by a factor of at least three. However, this is at the expense of samples size and measurement uncertainty, which increase. The uncertainty is estimated at <0.02 Fm to account for potential variability due to ongoing development of the system, but results are routinely better than 0.015 Fm. This is the outer limit that a scientist might reasonably expect their samples to fall within. An uncertainty of 0.02 Fm is equivalent to ±160 Ryr for a modern sample and limiting age of 31,400 Ryr. Conventional and reconnaissance dating methods can achieve high precision, low background results with samples down to a few mg of carbonate in size, but it is the rapid and low cost analyses that are offered by the CFAMS method that makes it a useful tool for reconnaissance surveys where sufficient sample exists. High precision is not guaranteed for the reconnaissance dating method as modern contamination and residual organic material can be incorporated during preparation of samples and bias radiocarbon values.

[1] Auxiliary materials are available in the HTML. doi:10.1029/2011PA002174.

Table 1. Summary Statistics for the Blank and Secondary Standards Used in This Study

Standard	Concensus ^{14}C Value (Fm)	^{14}C Value by AMS (Mean Fm ± 1σ)	^{14}C Value By CFAMS (Mean Fm ± 1σ)
IAEA C1 Marble	0 ± 0.0002	0.0019 ± 0.0004 (n = 8)	0.0090 ± 0.0031 (n = 23)
IAEA C2 Travertine	0.4114 ± 0.0003	0.4119 ± 0.0021 (n = 5)	0.4092 ± 0.0062 (n = 12)
UCI Coral Standard	0.9453 ± 0.0019	0.9408 ± 0.0038 (n = 10)	0.9463 ± 0.0054 (n = 3)

4.2. Apparatus

[21] The apparatus was designed to deliver a constant stream of CO_2 gas to the ion source and to analyze coral and other carbonate samples that were not limited by size. The ion source current is maximized with pure CO_2 and, not only is tuning facilitated with pure CO_2 but calibration is straightforward when the relative amount of CO_2 in the gas delivered to the ion source remains constant. Carbonate samples are well-suited to the generation of pure CO_2 gas by acidification, and the use of a syringe pump permits the precise control of the flow rate of gas to the open split and source. A carrier gas was not used to displace the CO_2 from the vacutainer as it exponentially dilutes the CO_2, compromising source tuning and complicating data reduction.

[22] For these analyses, 25–30 mg of $CaCO_3$ was used. Twenty-eight mg of $CaCO_3$ in the 7 ml vacutainer containing 1 mL of acid yielded 6 mL of pure CO_2. With the syringe pump set to deliver water to the vacutainer at 1 mL/min, we could record data for 5 min, leaving a small head space of CO_2 behind to prevent water entering the capillary. Smaller samples can be analyzed by reducing the flow rate of the syringe pump. For example, at 0.5 mL/min the amount of sample can be halved. The intake of the gas ion source of 0.37 mL/min limits the minimum size of the sample as the flow rate of the syringe pump must exceed the intake of the source. The data recording time and hence the amount of sample can be further reduced although this comes at the expense of precision. For example, 8 mg of $CaCO_3$ (1 mg C) would yield 2 mL of CO_2 from which 1 min of data could be recorded with increase in error of at least 2.2 times (i.e.$\sqrt{5}$). Continuing work on the source and delivery system is achieving further improvements in performance. As a next step to create a rapid, high throughput system, we plan to upgrade the system with a Gilson automated gas and liquid handling system.

4.3. Standards

[23] Table 1 gives the means data and standard deviation for data from the 3 analyses. An average blank (C1) of 0.0090 Fm (~37,800 Ryr) was achieved in this study and the C2 and CSTD secondary standards fell within consensus values. The errors on CFAMS data show how precision is degraded with this method. Background is primarily affected by the changeover time between samples. The ion source and delivery system have a memory due to flushing of the CO_2 gas that can be fitted with an exponential decay curve with a time constant of at least 2 s. When samples are changed over without delay (i.e., minimal backflushing) some carryover of reference gas into the C1 blank occurs. Additional time spent back flushing the system with argon can improve the background and we also found that the addition of a small amount of oxygen to the argon backflush for the source helped minimize carryover. The magnitude of the blank in a particular batch of samples ultimately determines the limiting age of the analysis and is subtracted from the sample data during background correction. To a lesser degree, the ion source development and sample preparation can degrade precision. Vacuum is occasionally compromised on the vacutainers which can reduce sample or standard integrity and affect data. While the reduced age range and precision is a trade-off of this method, it is more than acceptable for screening samples, conducting broad surveys and identifying trends.

4.4. Initial Survey

[24] The initial survey of 20 corals was part of a 65 sample batch including standards. This was analyzed within a 48 h period involving preparation one afternoon, analysis by CFAMS the next day, and data processing the following morning. Nowadays this is typically done in 1.5 days. Comparison of the data from the CFAMS system with data previously obtained using conventional methods showed a linear relationship with a correlation coefficient of 0.999. This indicated the methods were in agreement however the slope of the trend line was less than 1 which indicated the CFAMS data was slightly biased to older values. Possible reasons may include a systematic instrument bias or that the AMS samples had been analyzed several years prior using a different sampling or cleaning conditions. Whatever the case, this initial survey clearly demonstrated the viability and speed of the method, and that further tests were required for the assessment of performance. Using this survey data, we selected 5 corals with an evenly distributed age range between 0 and 1 Fm and reanalyzed them in triplicate with

Table 2. Comparison of Methods Used for Radiocarbon Dating of Carbonates at NOSAMS[a]

Parameter	Conventional AMS	Reconnaissance Dating	CFAMS
CO_2 preparation	Acid-Carbonate Reaction	Elemental Analyzer	Acid-Carbonate Reaction
Graphite Preparation	Conventional Graphitisation	Sealed Tube Graphitisation	N/A
Radiocarbon Measurement	AMS	AMS	GIS-AMS
Sample required (mg $CaCO_3$)	8–12	4–20	25–30
Time for 40 samples (days)[b]	5–6	3–4	1.5
Relative Cost	1	0.4	0.3
Mesurement Uncertainty (Fm)[c]	0.002–0.003	0.003–0.015	<0.02

[a]For specific details contact NOSAMS or visit www.nosams.whoi.edu.
[b]Typical laboratory turnaround times excluding the sample queue.
[c]Precision. For CFAMS this includes variability arising from system development.

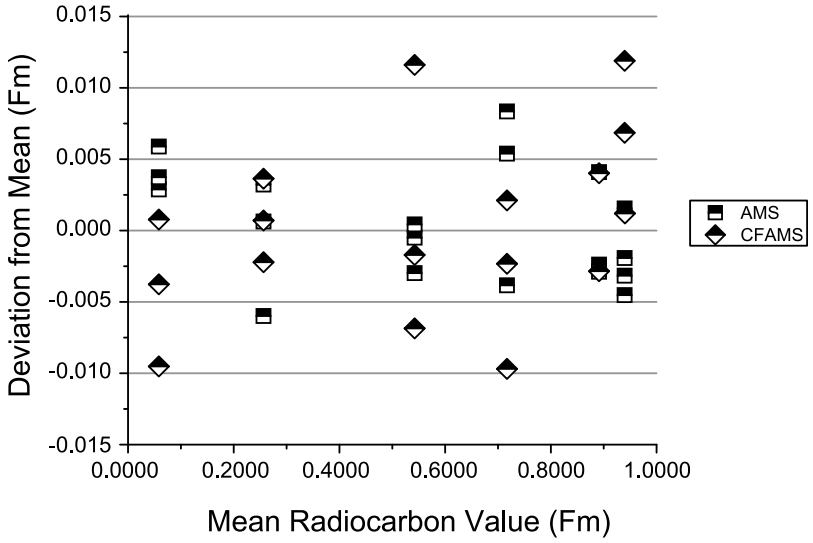

Figure 2. Analysis of 5 selected corals plus the coral standard in triplicate by CFAMS and conventional AMS. Deviation from mean versus the mean Fm of each coral where mean is the average of all Fm values from both methods.

the CSTD coral. We prepared all the samples identically for this test to ensure that there was no bias from sampling or cleaning.

4.5. Triplicate Analysis

[25] As with the initial survey, the repeat analysis of the samples with the CFAMS method was able to be completed in less than 2 days. Data from both methods was in good agreement with a slope of 1.008 ± 0.008 for the weighted double error least squares fit and a correlation coefficient of 0.999. The results of the t-test using the replicate data for each coral and assuming unequal variance gave p-values greater than 0.05 indicating that there was no statistically significant difference between the methods. A mean - difference plot was constructed and shows that while the CFAMS method has a larger spread than the conventional AMS method but that there is no systematic bias to the values (Figure 2). All CFAMS values except 2 lie within confidence limits of 2 standard deviations (±0.0102 Fm) from the mean value and the largest deviation was 0.0119 Fm. This is comparable with the precision of the reconnaissance dating method of <0.015Fm and shows that the CFAMS method is similarly able to make radiocarbon measurement for reconnaissance surveys with reasonable accuracy and precision.

[26] The CFAMS data from both the initial and triplicate analyses were also analyzed using weighted double error least squares fit. This gave a slope of 1 and a correlation coefficient of 0.999 which showed that the sample data for CFAMS method were repeatable between batches. For the triplicate analysis, the error for the 2 modern corals was ± 0.006 (50 Ryr) and the oldest coral had a mean age of 0.054 ± 0.005 Fm (23, 500 ± 800 Ryr) showing data were repeatable within batch.

4.6. Sample Analysis

[27] The coral samples from the Galapagos were collected for paleoclimate studies. Here the CFAMS method was used to identify which samples would be of most value for these further studies. In this case, the ages of the corals fell into two groups that were >30,000 Ryr (n = 16) and <1,500 Ryr (n = 25) (Table S3). Evidently these corals will be of value for examining recent paleoceanography (<1,500 Ryr) and, with U-Th dating will be of value for examining the history of the region before the last glacial maximum. The analytical strategy was successful in giving rapid information on the age distribution of the corals and prevented time and resources from being wasted on less efficient techniques for age determination.

5. Conclusions

[28] We have successfully developed an apparatus for the rapid radiocarbon analysis of carbonates using corals. Results are sufficiently accurate, precise and repeatable for reconnaissance studies, surveys, and paleoecological studies where large numbers of samples need to be analyzed with moderate precision and background. The technique has achieved a further increase in speed and cost effectiveness that will open up new lines of enquiry in many disciplines of paleoceanography and sedimentology. We were able to cost effectively obtain an age distribution for corals from the Eastern Pacific Ocean and minimize sampling risk for further analysis in under 2 days.

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